

Minimum Heat of Formation of Potassium Iodo Hydride

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It was previously reported [R. Mills, B. Dhandapani, N. Greenig, J. He, "Synthesis and Characterization of Potassium Iodo Hydride", Int. J. of Hydrogen Energy, Vol. 25, Issue 12, December, (2000), pp. 1185-1203.] that a novel inorganic hydride compound *KHI* which comprised a high binding energy hydride ion was synthesized by reaction of atomic hydrogen with potassium metal and potassium iodide. Potassium iodo hydride was identified by time of flight secondary ion mass spectroscopy, X-ray photoelectron spectroscopy, ^1H and ^{39}K nuclear magnetic resonance spectroscopy, Fourier transform infrared spectroscopy, electrospray ionization time of flight mass spectroscopy, liquid chromatography/mass spectroscopy, thermal decomposition with analysis by gas chromatography, and mass spectroscopy, and elemental analysis. We report measurements of heats of formation of *KHI* by differential scanning calorimetry (DSC) on a very reliable commercial instrument, a Setaram HT 1000 DSC. With reactant *KI* present, potassium metal catalyst and atomic hydrogen were produced by decomposition of *KH* at an extremely slow rate under a helium atmosphere to increase the amount of atomic hydrogen by slowing the rate of molecular hydrogen formation. Since not all of the starting materials reacted, the observed minimum heats of formation were over $-2000 \text{ kJ/mole } \text{H}_2$ compared to the enthalpy of combustion of hydrogen of $-241.8 \text{ kJ/mole } \text{H}_2$.

INTRODUCTION

Based on the solution of a Schrödinger-type wave equation with a nonradiative boundary condition based on Maxwell's equations, Mills [1-36] predicts that atomic hydrogen may undergo a catalytic reaction with certain atomized elements or certain gaseous ions which singly or multiply ionize at integer multiples of the potential energy of atomic hydrogen, 27.2 eV , $m \cdot 27.28\text{ eV}$ wherein m is a integer. One such atomic catalytic system involves potassium metal. The first, second, and third ionization energies of potassium are 4.34066 eV , 31.63 eV , 45.806 eV , respectively [37]. The triple ionization ($t=3$) reaction of K to K^{3+} , then, has a net enthalpy of reaction of 81.7766 eV , which is equivalent to $3 \cdot 27.2\text{ eV}$. Potassium ions can also provide a net enthalpy of a multiple of that of the potential energy of the hydrogen atom. The second ionization energy of potassium is 31.63 eV ; and K^+ releases 4.34 eV when it is reduced to K [37]. The combination of reactions K^+ to K^{2+} and K^+ to K , then, has a net enthalpy of reaction of 27.28 eV , which is equivalent to $1 \cdot 27.2\text{ eV}$.

Observation of intense extreme ultraviolet (EUV) emission at low temperatures (e.g. $\approx 10^3\text{ K}$) from atomic hydrogen and certain atomized elements or certain gaseous ions [6-21] has been reported previously. The only pure elements that were observed to emit EUV were those wherein the ionization of t electrons from an atom to a continuum energy level is such that the sum of the ionization energies of the t electrons is approximately $m \cdot 27.2\text{ eV}$ where t and m are each an integer. Potassium, cesium, and strontium atoms and Rb^+ ion ionize at integer multiples of the potential energy of atomic hydrogen and caused emission. Whereas, the chemically similar atoms, sodium, magnesium and barium, do not ionize at integer multiples of the potential energy of atomic hydrogen and caused no emission. Additional prior studies that support the possibility of a novel reaction of atomic hydrogen which produces an anomalous discharge and produces novel hydride compounds [6-35] include: 1.) the recent analysis of mobility and spectroscopy data of individual electrons in liquid helium which shows direct experimental confirmation that electrons may have fractional principal quantum energy levels [36], 2.) the observation of continuum state emission of Cs^{2+} and Ar^{2+} at 53.3 nm

and 45.6 nm, respectively, with the absence of the other corresponding Rydberg series of lines from these species which confirmed the resonant nonradiative energy transfer of 27.2 eV from atomic hydrogen to the catalysts atomic cesium or Ar^+ [6], 3.) the spectroscopic observation of the predicted hydride ion $H^-(1/2)$ of hydrogen catalysis by either cesium atom or Ar^+ catalyst at 407 nm corresponding to its predicted binding energy of 3.05 eV [6], 4.) the identification of transitions of atomic hydrogen to lower energy levels corresponding to lower energy hydrogen atoms in the extreme ultraviolet emission spectrum from interstellar medium [1, 5], 5.) the EUV spectroscopic observation of lines by the Institut Fur Niedertemperatur-Plasmaphysik e.V. that could be assigned to transitions of atomic hydrogen to lower energy levels corresponding to lower energy hydrogen atoms and the emission from the excitation of the corresponding hydride ions [11], 6.) the observation by the Institut Fur Niedertemperatur-Plasmaphysik e.V. of an anomalous plasma and plasma afterglow duration formed with hydrogen-potassium mixtures [12], 7.) the observation of anomalous afterglow durations of plasmas formed by catalysts providing a net enthalpy of reaction within thermal energies of $m \cdot 27.28$ eV [12-13], 8.) the observation of Lyman series in the EUV that represents an energy release 10 times hydrogen combustion which is greater than that of any known chemical reaction [6-21], 9.) the observation of line emission by the Institut Fur Niedertemperatur-Plasmaphysik e.V. with a 4 ° grazing incidence EUV spectrometer that was 100 times more energetic than the combustion of hydrogen [11], 10.) the observation of anomalous plasmas formed with strontium and argon catalysts at 1% of the theoretical or prior known voltage requirement with a light output for power input up to 8600 times that of the control standard light source [8-9, 14], 11.) the observation that the optically measured output power of gas cells for power supplied to the glow discharge increased by over two orders of magnitude depending on the presence of less than 1% partial pressure of certain of catalysts in hydrogen gas or argon-hydrogen gas mixtures [7], 12.) the isolation of novel hydrogen compounds as products of the reaction of atomic hydrogen with atoms and ions which formed an anomalous plasma as reported in the EUV studies [17, 19-35], 13.) the identification of novel hydride compounds by i.) time of flight secondary ion mass spectroscopy

which showed a dominant hydride ion in the negative ion spectrum, ii.) X-ray photoelectron spectroscopy which showed novel hydride peaks and significant shifts of the core levels of the primary elements bound to the novel hydride ions, iii.) ^1H nuclear magnetic resonance spectroscopy (NMR) which showed extraordinary upfield chemical shifts compared to the NMR of the corresponding ordinary hydrides, and iv.) thermal decomposition with analysis by gas chromatography, and mass spectroscopy which identified the compounds as hydrides [17, 19-35], 14.) the NMR identification of novel hydride compounds MH^*X wherein M is the metal, X , is a halide, and H^* comprises a novel high binding energy hydride ion identified by a large distinct upfield resonance [22-23, 26], 15.) the replication of the NMR results of the identification of novel hydride compounds by large distinct upfield resonances at Spectral Data Services, University of Massachusetts Amherst, University of Delaware, Grace Davison, and National Research Council of Canada [22], 16.) the NMR identification of novel hydride compounds MH^* and MH_2^* wherein M is the metal and H^* comprises a novel high binding energy hydride ion identified by a large distinct upfield resonance that proves the hydride ion is different from the hydride ion of the corresponding known compound of the same composition [22].

A novel inorganic hydride compound KHI which comprises a high binding energy hydride ion was previously synthesized by a catalytic reaction of atomic hydrogen with potassium metal and potassium iodide [22-23, 26]. Potassium iodo hydride was prepared in a stainless steel gas cell comprising a Ti screen hydrogen dissociator and KI . Potassium metal was added to the cell under an argon atmosphere. The cell was then continuously evacuated with a high vacuum turbo pump and heated to 650 °C. The hydrogen pressure was then maintained at 1500 torr for 48 hours after which time the cell was cooled, and blue crystals of the KHI product were removed and analyzed. The synthesis was repeated with the exception that the hydrogen was slowly added to maintain a pressure within the range of 1 torr to 10 torr and green crystals of KHI formed.

The reported [23] time of flight secondary ion mass spectroscopy (ToF-SIMS), X-ray photoelectron spectroscopy (XPS), ^1H and ^{39}K nuclear magnetic resonance spectroscopy (^1H and ^{39}K NMR), Fourier transform infrared spectroscopy (FTIR), electrospray ionization time of flight mass

spectroscopy (ESITOFMS), liquid chromatography/mass spectroscopy (LC/MS), thermal decomposition with analysis by gas chromatography (GC), and mass spectroscopy (MS), and elemental analysis results confirm the identification of *KHI* having hydride ions, $H^-(1/2) E_b = 3.05 \text{ eV}$, $H^-(1/4) E_b = 11.2 \text{ eV}$, and $H^-(1/6) E_b = 22.8 \text{ eV}$ predicted by Mills [1]. Two forms of hydride ion ($H^-(1/2)$ and $H^-(1/4)$) may be formed with two potassium ions or potassium metal as the catalyst, respectively, which was supported by the XPS, NMR, and LC/MS data of the blue crystals. The ToF-SIMS, XPS, and NMR results of the green crystals indicated that a higher binding energy hydride $H^-(1/6)$ formed by running the catalysis reaction at lower hydrogen pressure.

The LC/MS data indicated that the blue crystal comprised a novel compound *KHI* which contained two different hydride ions that gave rise to different mass fragmentation patterns. One *KHI* compound with a retention time of $RT = 11.42 \text{ min.}$ gave rise to a $K(KI)_2^+$ mass fragment. Whereas, a second *KHI* compound with a retention of about $RT = 23 \text{ min.}$ gave rise to a $K(KI)^+$ and a $K(KI)_2^+$ mass fragment.

The positive ion ESITOFMS spectrum of the blue crystals and that of the *KI* control were dominated by the K^+ ion. A series of positive ions $K[KI]_n^+$ $m/z = (39 + 166n)$ were also observed. In addition, KHI^+ was only observed from the blue crystals.

The positive and negative ToF-SIMS results of the blue crystals were consistent with the proposed structure *KHI*. The positive ion spectrum of the blue crystals and that of the *KI* control were dominated by the K^+ ion. The comparison of the positive ToF-SIMS spectrum of the *KI* control with the blue crystals demonstrated that the $^{39}K^+$ peak of the blue crystals saturated the detector and gave rise to a peak that was atypical of the natural abundance of ^{41}K which was indicative of a unique crystalline matrix. The negative ion ToF-SIMS of the blue crystals was dominated by H^- and I^- peaks of about equal intensity. Iodide alone dominated the negative ion ToF-SIMS of the *KI* control.

The positive and negative ToF-SIMS spectrum obtained from the green crystals was similar to that obtained from the blue crystals except that the hydride ion peak of the negative ToF-SIMS spectrum obtained from the green crystals was much larger than that obtained from the blue crystals. This result was consistent with the formation of a higher

hydride content or a higher hydride ion yield in the green crystals which indicated that a higher binding energy hydride was formed by running the catalysis reaction at lower hydrogen pressure.

The XPS data of the core levels clearly indicated a change in the electronic structure and different bonding in *KHI* relative to that in the corresponding *KI*. This binding influenced the metal core level with little perturbation of the halogen core level. Comparing the alterations in the *K* core levels versus the *I* core level indicated that the lower-energy hydrogen species binds to the metal center of *KI*. An additional spin-orbit splitting component had to be added to each potassium iodo hydride in order to obtain a good curve fit of the *K 2p* spectra. In each case, the second component of spin-orbit splitting was assigned to the formation of the alkali metal halido hydride, *KHI*. The presence of the novel hydride ion shifted the *K 2p* peaks to lower binding energies relative to the corresponding peaks of *KI*. The change in electronic structure was greatest with the green crystals which indicates that a higher binding energy hydride was formed by running the catalysis reaction at lower hydrogen pressure.

The 0-100 eV binding energy region of a high resolution XPS spectra of the blue and green crystals indicated the presence of the hydride ions $H^-(1/2)$ and $H^-(1/4)$ in the case of the blue crystals and the presence of the higher binding energy hydride ion $H^-(1/6)$ in the case of the green crystals. This product was predicted by an autocatalysis reaction of two $H(1/4)$ atoms which has been confirmed by extreme ultraviolet spectroscopy [11].

The upfield peak in the NMR spectrum of the green crystals at -2.5 ppm was assigned to a novel hydride ion that has a smaller radius than that of the hydride ions observed in the case of the blue crystals corresponding to resonances at -0.376 ppm and -1.209 ppm since the upfield shift was larger in the case of the green crystals. A smaller radius corresponds to a higher binding energy. The NMR results of the identification of *KHI* by large distinct upfield resonances have been reproduced at Spectral Data Services, University of Delaware, Grace Davison, and National Research Council of Canada [22].

In the present study, the heats of formation of *KHI* was measured by differential scanning calorimetry (DSC) on a very reliable commercial

instrument, a Setaram HT 1000 DSC. With reactant *KI* present, potassium metal catalyst and atomic hydrogen were produced by decomposition of *KH* at an extremely slow rate under a helium atmosphere to increase the amount of atomic hydrogen by slowing the rate of molecular hydrogen formation. MgH_2 was run as a control since no ionization reaction is possible for magnesium metal or magnesium ions with a net positive enthalpy of an integer multiple of the potential energy of atomic hydrogen, $m \cdot 27.28 \text{ eV}$. No emission was observed for magnesium in previous EUV and visible spectroscopy studies of vaporized magnesium metal with atomic hydrogen [6-10]. As further controls to test the dependence of the on the reaction on atomic hydrogen, a hydrogen atmosphere replaced the helium atmosphere, or the heating ramp rate was increased.

EXPERIMENTAL

Potassium hydride (*KH*, Aldrich, 30 wt. % dispersion in mineral oil, Catalog #21,581-3, Lot # E107116JU) was cleaned by washing with hexane. The *KH*/ mineral oil suspension was allowed to settle inside the glove box. The layer of mineral oil was separated from the *KH* layer. 10 g of the unwashed *KH* containing mineral oil was placed in a 100 ml Schlenk tube. The Schlenk tube was connected to a vacuum manifold outside the glove box. 20 ml of hexane (Aldrich, anhydrous, Catalog # 22,706-4, used as supplied by manufacturer) was added under argon flow. After stirring for 5 minutes, the *KH* was allowed to settle, and the supernatant liquid was removed with a syringe. The hexane washing procedure was repeated 4 additional times. The *KH* was dried under vacuum for 3 hours and stored in a glove box prior to use.

Differential scanning calorimeter (DSC) measurements were performed using the DSC mode of a Setaram HT-1000 calorimeter (Setaram, France). Two matched Alumina glove fingers were used as the sample compartment and the reference compartment. The fingers permitted the control of the reaction atmosphere. 0.050 g of washed *KH* was placed in a flat-base Al-23 crucible (Alfa-Aesar, 15 mm high x 10 mm OD x 8 mm ID) and covered with 0.500 g of *KI* (Alfa-Aesar, 99.99%). The crucible was then placed in the bottom of the sample Alumina glove

finger cell. As a reference, an aluminum oxide sample (Alfa-Aesar, -400 Mesh powder, 99.9%) with similar weight as the sample was placed in a matched Al-23 crucible. All samples were handled in a dry box. Each Alumina glove finger cell was sealed in the glove box, removed from the glove box, and then quickly attached to the Setaram calorimeter. The system was immediately evacuated to pressure of 1 mtorr or less. The cell was back filled with one atmosphere of helium and evacuated again. The cell was then refilled with helium to 760 torr. The cells were then inserted into the oven, and secured to their positions in the DSC instrument. The oven temperature was brought to the desired starting temperature of 100 °C. The oven temperature was scanned from 100 °C to 750 °C at a ramp rate of 0.1 degree/minute.

As a control, one atmosphere of hydrogen replaced the one atmosphere of helium. As a second control, MgH_2 replaced KH and KI . A 50 mg MgH_2 sample (Alfa-Aesar, 90%, reminder Mg) was added to the sample cell, while a similar weight of aluminum oxide (Alfa-Aesar) was added to the reference cell. Both samples were also handled in a dry box. As a third control, the ramp rate was doubled to 0.2 degree/minute for the DSC of the mixture of KH and KI . As a fourth control with 0.500 g of KI and 760 torr helium, 0.050 g of potassium metal replaced the 0.050 g of KH .

A DSC of a sample comprising a majority KI and containing some KHI synthesized previously [23] and a DSC of KI were run in order assist in the identification of the reaction product which may have occurred during the DSC experiment.

RESULTS AND DISCUSSION

The DSC (100-750 °C) results of KH and KI mixtures are shown in Figures 1-2. In Figure 1, a broad endotherm was observed at 300 to 400 °C which corresponded to 105.2 kJ/mole H_2 . Potassium hydride decomposes in this temperature range (288 to 415 °C) with a corresponding enthalpy of 118.2 kJ/mole H_2 [38]. A large exotherm was observed in the region 435 to 675 °C which corresponded to -2306 kJ/mole H_2 . A feature was observed within the exotherm at 410 to 475 °C that may correspond to an endothermic component due to the vaporization of potassium metal. The

boiling point of potassium metal is 756.5 °C with a corresponding enthalpy of vaporization of 81.1 kJ/mole $K(m)$ [39]. Another endotherm within the exothermic feature was observed at 673 °C. This is close to the known melting point of KI of 681 °C with a corresponding enthalpy of fusion of 24.0 kJ/mole KI [40]. The difference in temperatures may be due to the presence of KHI product. A DSC of a sample from the KHI synthesis reported previously [23] is shown in Figure 3. The same endothermic feature was observed at 673 °C corresponding to an enthalpy of 16.84 kJ/mole KHI as well as a new feature at 537 °C corresponding to an enthalpy of 1.52 kJ/mole KHI . The DSC of pure KI is shown in Figure 4. Only a sharp endotherm at 681 °C corresponding to an enthalpy of 23.90 kJ/mole KI was observed. The control KI DSC results closely matched the published melting point and heat of fusion of KI [40].

In repeat DSC experiments of KH and KI mixtures, the heat balance features were very similar. In Figure 2, a broad endotherm was observed at 300 to 400 °C which corresponded to 90.4 kJ/mole H_2 . A large exotherm was observed in the region 435 to 675 °C which corresponded to -2332 kJ/mole H_2 . Within the exotherm the endothermic components at 410 to 475 °C and 673 °C were also observed.

Flames from potassium metal were observed upon exposure of the contents of the sample glove finger to air following the DSC run; whereas, KHI was reported to be nonflammable [23]. Since not all of the starting materials reacted, the observed minimum heats of formation of KHI were -2306 kJ/mole H_2 compared to the enthalpy of combustion of hydrogen of -241.8 kJ/mole H_2 [41].

The DSC (100-750 °C) results of MgH_2 are shown in Figures 5-6. In both cases, two sharp endothermic peaks were observed. In Figures 5 and 6, a first peak was observed centered at 351.75 °C corresponding to 68.61 kJ/mole MgH_2 and at 368.52 °C corresponding to 59.11 kJ/mole MgH_2 , respectively. The decomposition of MgH_2 is observed at 440 to 560 °C corresponding to 74.4 kJ/mole MgH_2 [38]. In Figures 5 and 6, a second peak was observed centered at 648.46 °C corresponding to 6.65 kJ/mole MgH_2 and at 648.32 °C corresponding to 5.10 kJ/mole MgH_2 , respectively. The known melting point of $Mg(m)$ is 650 °C corresponding to an enthalpy of fusion of 8.48 kJ/mole $Mg(m)$ [42].

When the DSC reactions of *KH* and *KI* mixtures were repeated with one atmosphere of hydrogen replacing helium, only the known endothermic peaks of *KH* decomposition and *KI* melting were observed. The same result was observed when the heating rate was changed from 0.1 degree/minute to 0.2 degree/minute. The addition of an inert gas such as helium decreases the rate decomposition of a hydride by reducing the rate of molecular hydrogen formation from atomic hydrogen [43]. The rate of molecular hydrogen formation from atomic hydrogen is also decreased by decreasing the partial pressure of atomic hydrogen [44]. The partial pressure of atomic hydrogen is dependent on the hydride decomposition rate. In addition, when MgH_2 replaced *KH*, only the known endothermic peaks of MgH_2 decomposition and $Mg(m)$ melting were observed. No exotherm was observed for the control with 0.500 g of *KI*, 760 torr helium, and 0.050 g of potassium metal which replaced the 0.050 g of *KH*. The results indicate that the exothermic reaction requires atomic hydrogen and potassium metal which supports the catalysis of atomic hydrogen by potassium atoms as the source of the large exotherm. These results are consistent with the previous reports of the formation of an anomalous plasma with atomic hydrogen and potassium but not with magnesium [6-10].

CONCLUSION

The observed minimum heats of formation of *KHI* by the catalytic reaction of potassium with atomic hydrogen and *KI* were over $-2000 \text{ kJ/mole } H_2$ compared to the enthalpy of combustion of hydrogen of $-241.8 \text{ kJ/mole } H_2$. Since the net enthalpy released is essentially an order of magnitude that of combustion, the catalysis of atomic hydrogen represents a new source of energy with H_2O as the source of hydrogen fuel. Moreover, rather than air pollutants or radioactive waste, novel hydride compounds with potential commercial applications are the products [45-46].

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Figure Captions

- Figure 1. The results of the DSC (100-750 °C) of *KH* and *KI* at a scan rate of 0.1 degree/minute.
- Figure 2. The repeat results of the DSC (100-750 °C) of *KH* and *KI* at a scan rate of 0.1 degree/minute.
- Figure 3. The results of the DSC (100-750 °C) of *KI* with some *KHI* at a scan rate of 0.1 degree/minute.
- Figure 4. The results of the DSC (100-750 °C) of *KI* at a scan rate of 0.1 degree/minute.
- Figure 5. The results of the DSC (100-750 °C) of MgH_2 at a scan rate of 0.1 degree/minute.
- Figure 6. The repeat results of the DSC (100-750 °C) of MgH_2 at a scan rate of 0.1 degree/minute.

Fig. 1

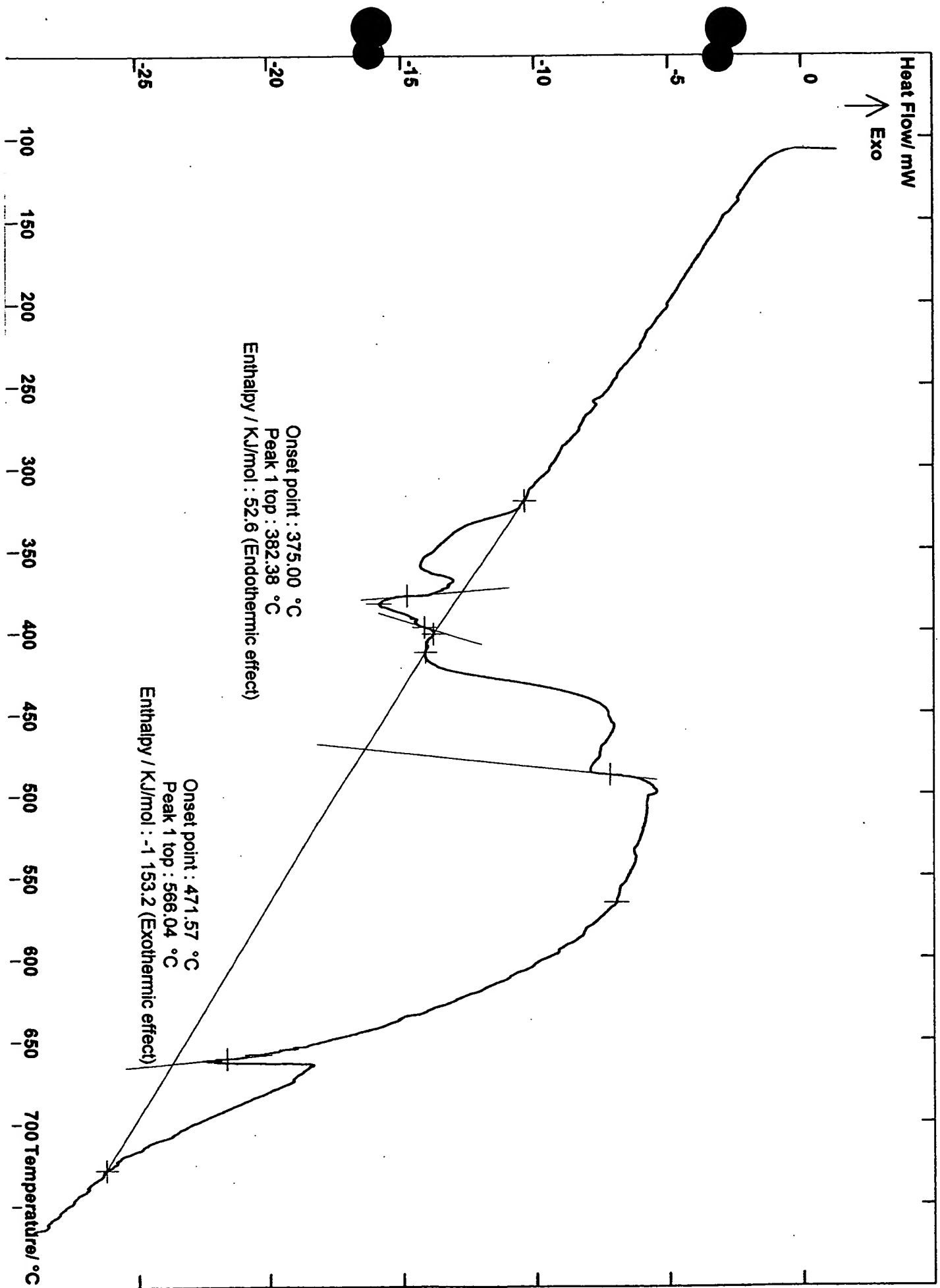


Fig. 2

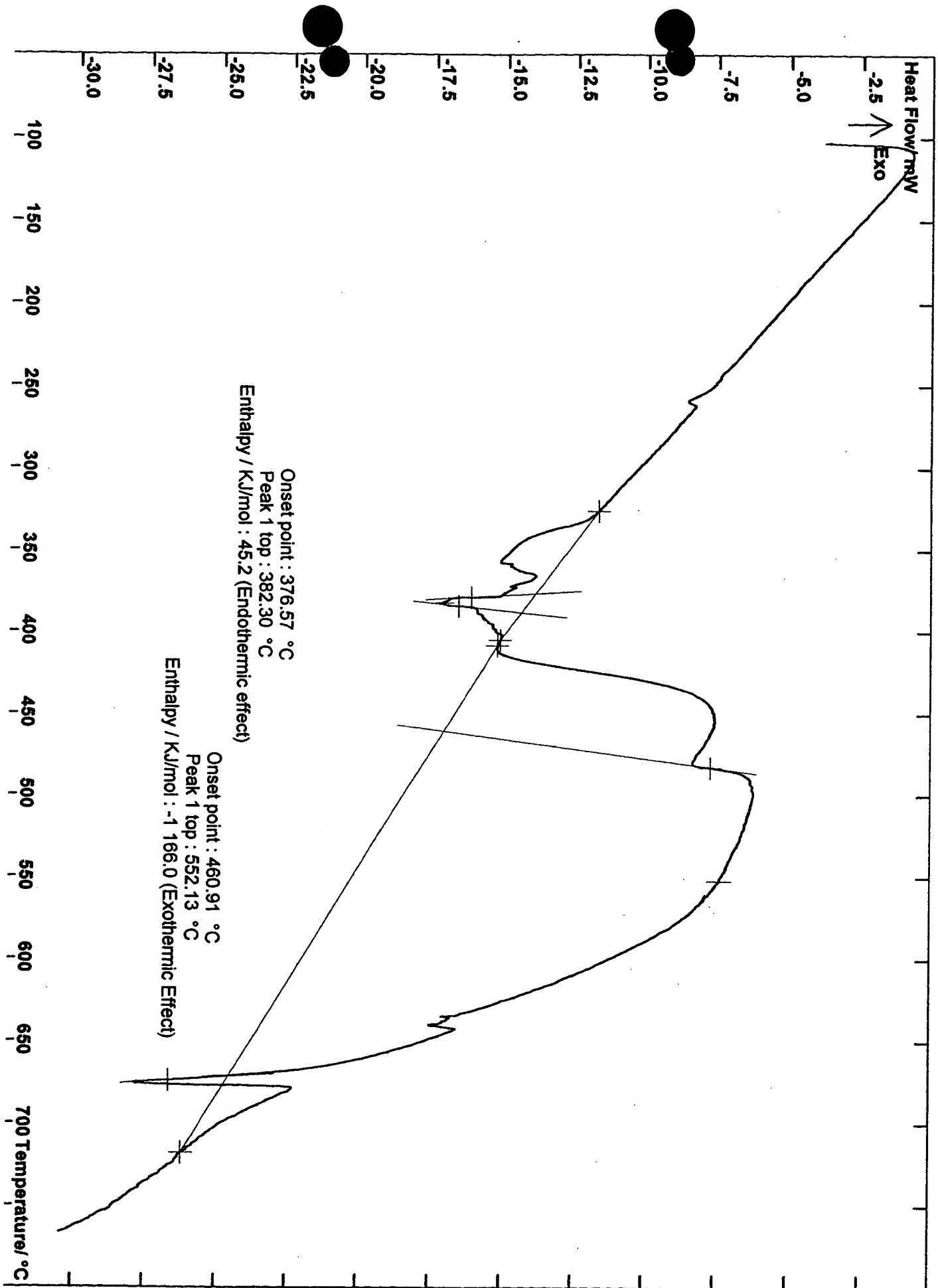


Fig. 3

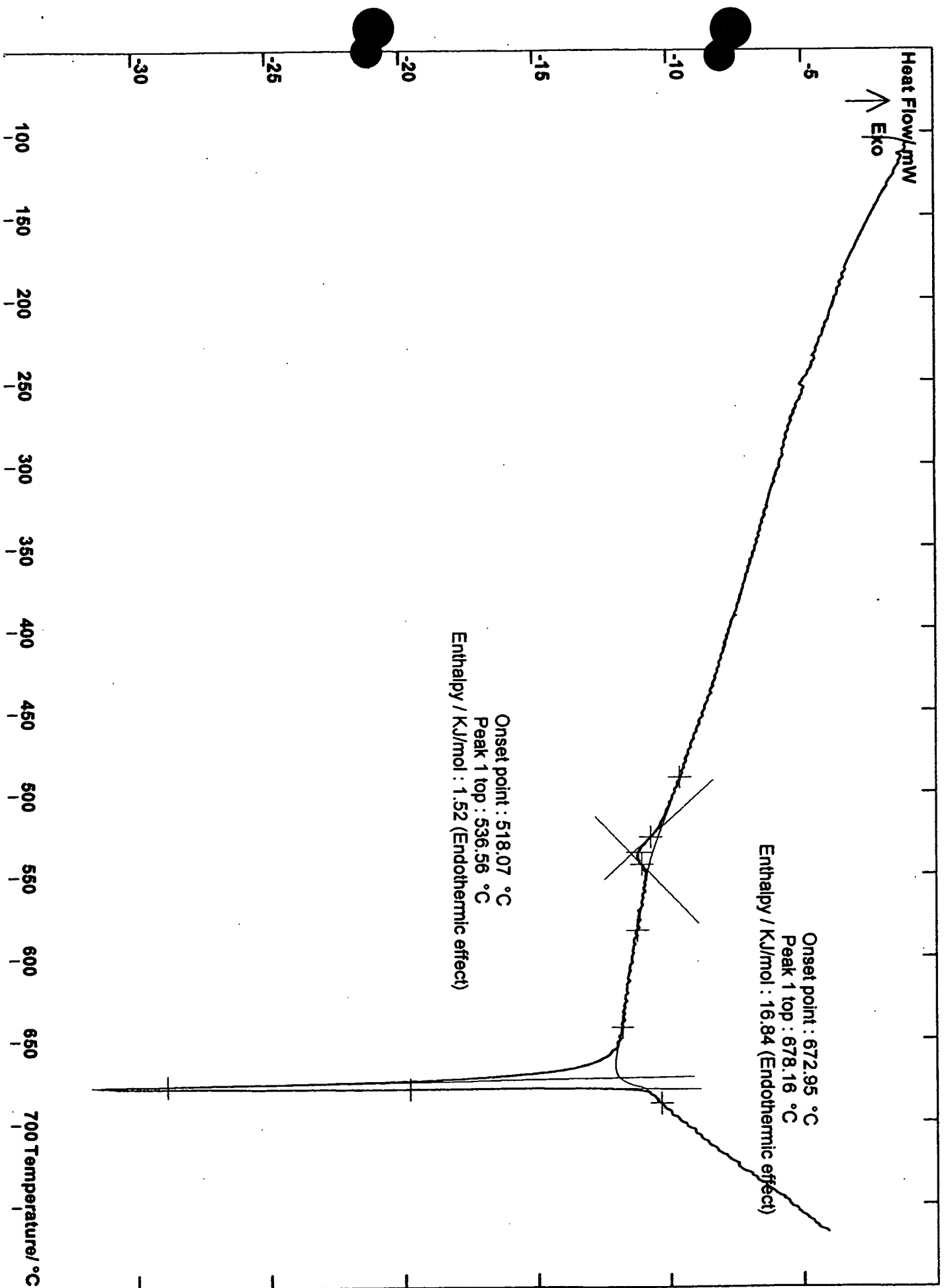


Fig. 4

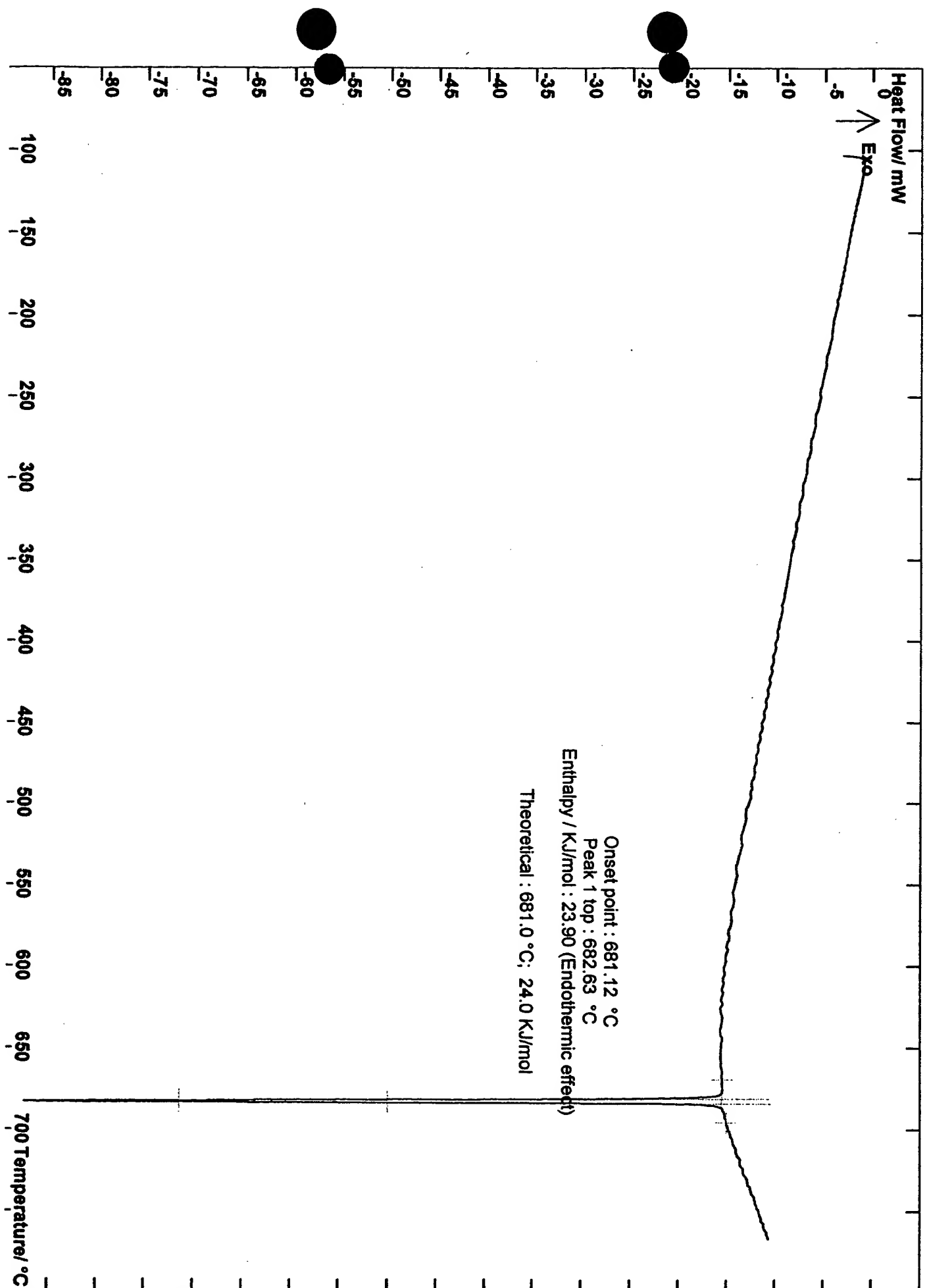


Fig. 5

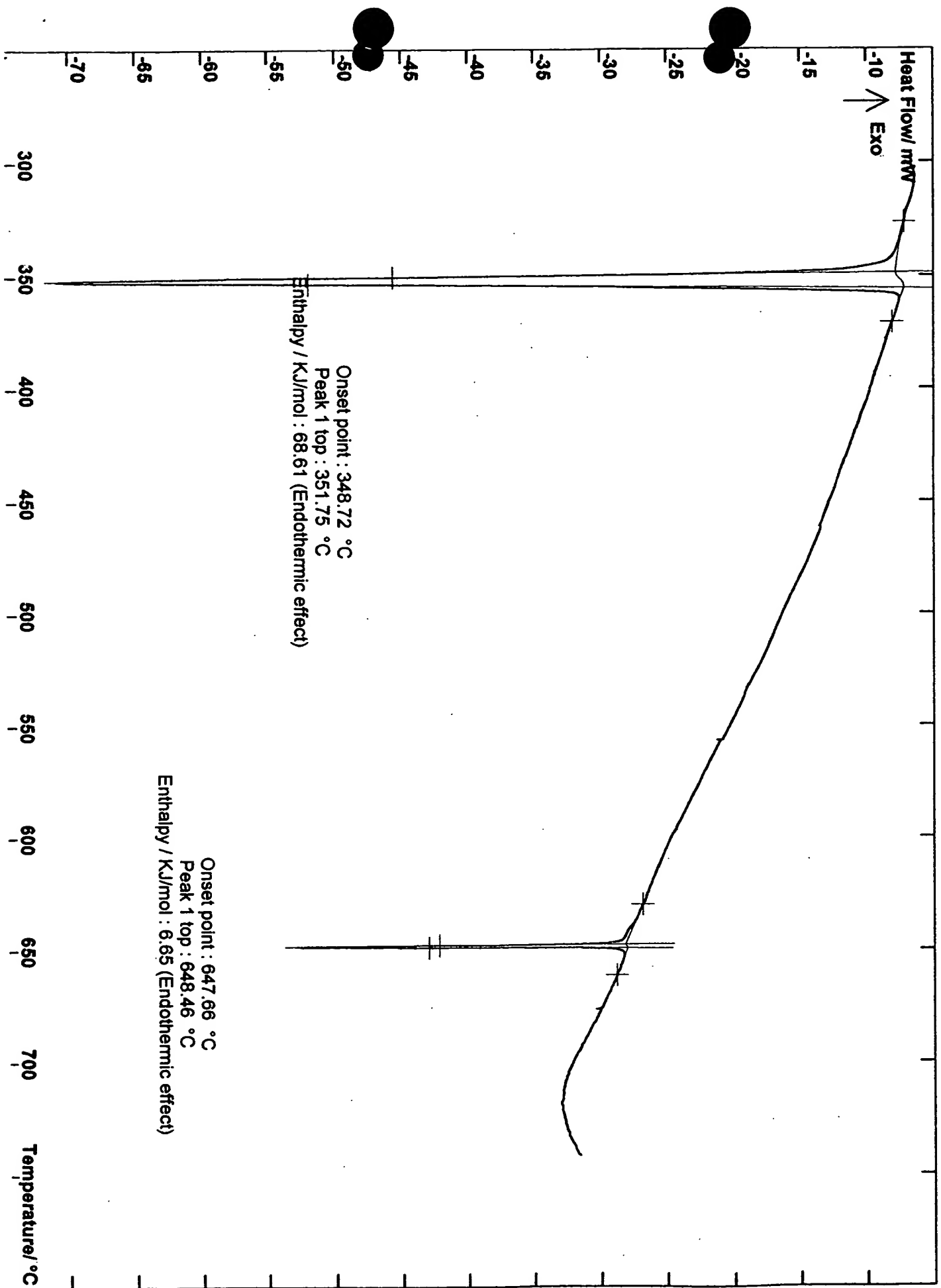


Fig. 6

